INTERNATIONAL STANDARD 473

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Lithopone pigments for paints

Lithopone pour peintures

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Lithopone pigments for paints

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the requirements and the corresponding methods of test for two types of lithopone, suitable for use in paints and related products.

2 REFERENCES

ISO 787, *General methods of test for pigments.*

ISO 842, *Raw materials for paints and varnishes – Sampling.*

3 DEFINITIONS

3.1 lithopone 30 % : A white pigment consisting of zinc sulphide (ZnS) and barium sulphate ($BaSO_4$) in approximately equimolecular proportions. The material is a calcined co-precipitate.

3.2 lithopone 60 %[1] : A white pigment which contains approximately 60 % zinc sulphide (ZnS) and a balance consisting mainly of barium sulphate ($BaSO_4$). The material is a calcined co-precipitate.

4 REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

Lithopone pigments for paints shall have the characteristics shown in the table.

5 SAMPLING

A representative sample of the pigment shall be taken in accordance with ISO 842.

TABLE – **Required characteristics and their tolerances**

Characteristic	**Specification**		**Test method**
	lithopone 30 %	**lithopone 60 %**	
Total zinc, calculated as zinc sulphide % (*m/m*)	min. 28	min. 58	Clause 6
Zinc oxide % (*m/m*)	max. 1	max. 1	Clause 7
Sum of total zinc, calculated as zinc sulphide, and barium sulphate % (*m/m*)	min. 99	min. 99	Clause 6
Barytes	absent	absent	Clause 8
Matter volatile at 105 °C % (*m/m*)	max. 0,5	max. 0,5	ISO 787, Part II
Matter soluble in water % (*m/m*)	max. 0,5	max. 0,5	ISO 787, Part III
Alkalinity of the aqueous extract	Shall be equal to an agreed reference sample		ISO 787, Part IV
Residue on sieve (63 μm) % (*m/m*)	max. 0,1	max. 0,1	ISO 787, Part VI or Part VII
Colour	Shall be equal to that of an agreed reference sample		ISO 787, Part I
Lightening power	*To be agreed between the interested parties*		ISO 787, Part XVII
Oil absorption value			ISO 787, Part V
Hiding power			To be agreed

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[1] There are on the market

a) lithopones with a content of about 40 or 50 % of zinc sulphide. These products should be marked so as to indicate the zinc sulphide content;

b) barytes-reduced lithopones, which consist of mixtures of lithopones with higher zinc sulphide content and ground mineral barytes. These products should be marked so as to indicate the presence of barytes.

METHODS OF TEST

During the analysis, use only reagents of recognized analytical reagent grade, and only distilled water or water of equivalent purity.

6 DETERMINATION OF BARIUM SULPHATE AND TOTAL ZINC CONTENT

6.1 Reagents

6.1.1 Potassium hexacyanoferrate(II),[1] standard volumetric solution, approximately 0,05 M (concentration T_1, expressed in grams of zinc per millilitre).

Dissolve 21,0 g of potassium hexacyanoferrate(II), 300 mg of potassium hexacyanoferrate(III)[2] and 2 g of anhydrous sodium carbonate (to stabilize the solution) in water and dilute with water to 1 000 ml in a one-mark volumetric flask.

6.1.2 Zinc chloride, standard solution, containing approximately 5 g of zinc per litre (concentration T_2, in grams of zinc per millilitre).

Weigh about 5 g of chemically pure zinc to the nearest 0,1 mg, dissolve in 300 ml of hydrochloric acid (6.1.3) and dilute the solution obtained with water to 1 000 ml in a one-mark volumetric flask.

6.1.3 Hydrochloric acid, 4 M solution.

6.1.4 Sulphuric acid, 2 M solution.

6.1.5 Ammonia solution, 0,9 g/ml.

6.1.6 Ammonia solution, 4 M.

6.1.7 Diphenylamine, 50 g/l ethanolic solution.

6.1.8 Congo paper.

6.1.9 Lead acetate paper.

6.2 Standardization of the potassium hexacyanoferrate(II) solution

Pipette 25,0 ml of the zinc chloride solution (6.1.2) into a flask and add ammonia solution (6.1.6) until a piece of Congo paper (6.1.8), touched onto the solution, just turns to a pure red colour. Then carefully neutralize the solution with hydrochloric acid solution (6.1.3) from a dropping bottle. Add a few drops in excess until the Congo paper turns to a lasting red-blue or blue-red colour (pH 1,5 to 3,0).

Make up to 150 ml with water, heat the solution to boiling, and add 10 drops of the diphenylamine solution (6.1.7).

Immediately titrate the solution with the potassium hexacyanoferrate(II) solution (6.1.1) until the colour turns to a lasting yellow or yellowish green.

Then back-titrate the solution with the zinc chloride solution (6.1.2) until the colour just turns to blue again.

Calculate the concentration T_1, expressed in grams of zinc per millilitre, of the potassium hexacyanoferrate(II) solution by the formula

$$T_1 = \frac{T_2 \, (25 + V_2)}{V_1}$$

where

T_2 is the concentration, in grams of zinc per millilitre, of the standard zinc chloride solution (6.1.2);

V_1 is the volume, in millilitres, of the potassium hexacyanoferrate(II) solution (6.1.1) required for the titration;

V_2 is the volume, in millilitres, of the standard zinc chloride solution (6.1.2) required for the back-titration.

6.3 Procedure

Weigh, to the nearest 0,1 mg, about 0,6 g of lithopone 30 % previously dried at 105 ± 2 °C or about 0,3 g of lithopone 60 % previously dried at 105 ± 2 °C into a beaker and add 25 ml of the hydrochloric acid solution (6.1.3). Immediately cover with a watch-glass and boil until the evolution of hydrogen sulphide has ceased (test with lead acetate paper (6.1.9) . Dilute with 100 ml of water, add 5 ml of the sulphuric acid solution (6.1.4) and boil the solution again.

Allow the precipitate to settle whilst hot and filter the supernatant solution through a fine filter paper. Transfer the precipitate to the filter and wash with hot water containing some sulphuric acid until a drop of the washings shows no reaction with the potassium hexacyanoferrate(II) solution (6.1.1). Fold the filter paper over the precipitate. Transfer it whilst still wet to a weighed porcelain crucible and ignite it in contact with air to constant mass. The residue is assumed to be barium sulphate.[3] Add a few drops of the sulphuric acid solution to the contents of the crucible. No trace of hydrogen sulphide should be noticeable; otherwise, the sulphuric acid must be driven off and the residue re-ignited.

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1) IUPAC name; formerly called potassium ferrocyanide, $K_4Fe(CN)_6$.

2) IUPAC name; formerly called potassium ferricyanide, $K_3Fe(CN)_6$.

3) If desired, the actual barium sulphate content can be determined by fusing the residue with potassium sodium carbonate and converting the barium carbonate into barium sulphate.

Combine the washings with the filtrate. Add a slight excess of the ammonia solution (6.1.5) (verify on Congo paper), followed by the hydrochloric acid solution from a dropping bottle until a small piece of Congo paper, touched onto the solution, just turns to a lasting red-blue or blue-red colour (pH 1,5 to 3,0).

If necessary, make up to 150 ml with water, heat the solution to boiling, add 10 drops of the diphenylamine solution (6.1.7) and immediately titrate the solution in a similar manner as specified for the standardization of the potassium hexacyanoferrate(II) solution in 6.2.

6.4 Expression of results

Calculate the barium sulphate content of the lithopone, as a percentage by mass, by the formula :

$$\frac{100\,m_2}{m_1}$$

Calculate the total zinc content of the lithopone, as a percentage by mass, calculated as zinc sulphide, by the formula :

$$1{,}490\,(T_1V_3 - T_2V_4)\,\frac{100}{m_1} = \frac{149}{m_1}\,(T_1V_3 - T_2V_4)$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the residue;

T_1 is the concentration, expressed in grams of zinc per millilitre, of the potassium hexacyanoferrate(II) solution (6.1.1);

T_2 is the concentration, in grams of zinc per millilitre, of the standard zinc chloride solution (6.1.2);

V_3 is the volume, in millilitres, of the potassium hexacyanoferrate(II) solution (6.1.1) required for the titration;

V_4 is the volume, in millilitres, of the standard zinc chloride solution (6.1.2) required for the back-titration.

7 DETERMINATION OF THE ZINC OXIDE CONTENT

7.1 Reagents

7.1.1 Ammonium chloride.

7.1.2 Potassium hexacyanoferrate(II), standard volumetric solution approximately 0,05 M (concentration T_1) prepared as specified in 6.1.1 and standardized as specified in 6.2.

7.1.3 Zinc chloride, standard solution containing approximately 5 g of zinc per litre (concentration T_2) prepared as specified in 6.1.2.

7.1.4 Hydrochloric acid, 4 M solution.

7.1.5 Ammonia solution, 4 M.

7.1.6 Diphenylamine, 50 g/l ethanolic solution.

7.1.7 Congo paper.

7.2 Procedure

Weigh, to the nearest 1 mg, about 10 g of the lithopone previously dried at 105 ± 2 °C into a 500 ml one-mark volumetric flask. Add 4 g of the ammonium chloride (7.1.1) and 100 ml of the ammonia solution (7.1.5). Allow the suspension to stand for 1 h in the cold, shaking at intervals. After this period make the suspension up to the mark with water, shake and filter through an absolutely dry filter and funnel. Discard the first 10 to 20 ml of the filtrate and collect the remainder in a dry beaker. Remove 250 ml of this filtrate to a beaker by means of a pipette and add to it 10 ml of the zinc chloride solution (7.1.3) followed by the hydrochloric acid solution (7.1.4) from a dropping bottle until a piece of the Congo paper (7.1.7), touched onto the solution, turns to a lasting red-blue or blue-red colour (pH 1,5 to 3,0).

Heat the solution to boiling, add 10 drops of the diphenylamine solution (7.1.6) and titrate the solution immediately as specified for the standardization of the potassium hexacyanoferrate(II) solution in 6.2.

7.3 Expression of results

Calculate the zinc oxide content of the lithopone, as a percentage by mass, by the formula :

$$2{,}490\,(T_1V_5 - T_2V_6 - 10\,T_2)\,\frac{100}{m_3}$$

$$= \frac{249}{m_3}\,(T_1V_5 - T_2V_6 - 10\,T_2)$$

where

m_3 is the mass, in grams, of the test portion;

T_1 is the concentration, expressed in grams of zinc per millilitre, of the potassium hexacyanoferrate(II) solution (7.1.2);

T_2 is the concentration, in grams of zinc per millilitre, of the standard zinc chloride solution (7.1.3);

V_5 is the volume, in millilitres, of the potassium hexacyanoferrate(II) solution (7.1.2) required for the titration;

V_6 is the volume, in millilitres, of the standard zinc chloride solution (7.1.3) required for the back-titration.

8 DETECTION OF BARYTES (MINERAL BARIUM SULPHATE)

Detection of barytes may be carried out with certainty, within certain limits, only in the following manner :